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Synthesis of Nickel Nanoparticles Using Polyol Process

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ABSTRACT

Nickel nanoparticles (NiNPs) have been synthesized by the reduction of nickel acetate, $Ni(CH_3COO)_2$, with polyethylene glycol 400(PEG 400) without adding any surfactant or capping agent. PEG 400 served as a reducing agent as well as a solvent. Rod like nickel nanoparticles were obtained by the reduction of nickel acetate with PEG 400 at 100°C followed by centrifugation, washed with acetone and upon drying in air at room temperature. The synthesized nickel nanoparticles have been characterized by X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), High Resolution Transmission Electron Microscopy (HRTEM) and Energy Dispersive X-ray analysis (EDAX) techniques. The average crystallite size of the nickel nanoparticles were calculated from the XRD pattern using Scherrer's formula. EDAX analysis confirmed the presence nickel nanoparticles. The effect of reaction time, reaction temperature and concentration of the chemical on the size of the nickel nanoparticles formed have also been studied.

Keywords: Polyol process, PEG 400, nickel nanoparticles, HRTEM, EDAX.

1. INTRODUCTION

The field of nanoparticle research covers a wide range of interest in the fields of chemistry, physics and materials science. Nickel nanoparticles can exist in two crystalline structures, *fcc* and *hcp*. Most of the nickel powders are ferromagnetic substances. Nickel nanoparticles are found more useful in catalysis, conducting inks,

ferrofluids and magnetic materials[1]. Nanostructured nickel particles have received more attention because of their magnetic properties, which make them suitable for applications in magnetic sensors, memory devices and biomolecular separations[2-8]. Control of the nanoscale morphology enables precise control of the properties of the end product. The particle size, morphology and composition can be manipulated to produce materials of different properties[9-10]. In this present work, nickel nanoparticles have been synthesized using the polyol[11] process without adding any surfactant or capping agents and the particles were analyzed using XRD, SEM, HRTEM and EDAX. The effect of reaction time, reaction temperature and the concentration of the chemicals over the size of the particles have been studied.

EXPERIMENTAL

In a typical nickel nanoparticle (NiNP) preparation, 2g of nickel acetate was dissolved in appropriate amount of PEG-400 in a round bottom flask and allowed to reflux. The colour of the solution changed to black indicated the formation of NiNPs. After that the resultant solution was cooled to room temperature. The residue was separated by centrifugation followed by washing with acetone and dried in air at room temperature.

The resulting NiNPs were further characterized by XRD, SEM, HRTEM and EDAX. In order to study the effect of reaction time, reaction temperature and concentration of the chemicals over the size of the NiNPs, the reactions were performed at different reaction conditions. In order to study the effect of the reaction time over the size of the particles, 2g nickel acetate and 50 ml PEG 400 were taken and a temperature of 220°C is maintained and the reactions were carried out at 4 different time durations viz., 4 hours, 5 hours, 6 hours and 7 hours. A set of reactions were carried out at different temperatures viz., 100°C, 150°C, 200°C, 220°C and 250°C for duration of 5 hours by taking 2g nickel acetate and 50ml PEG 400 to study the effect of reaction temperature over the size of the NiNPs. The reactions were also conducted by varying the concentration of the chemicals to study the effect of concentration of the chemicals over the size of the NiNPs. The experimental conditions are shown in Table 1. The obtained NiNPs were analyzed using Bruker model D_8 advance X-ray diffractometer using CuKα(λ=1.5405A°) radiation and the XRD pattern were used to calculate the crystallite size of the NiNPs using Scherrer's formula[12]. The morphology of the nickel nanoparticles were analyzed by Scanning Electron Microscopy (SEM). The size of the synthesized nickel nanoparticles were analysed by HRTEM. The elemental composition of the nanoparticles was estimated by Energy dispersive X-ray (EDAX) analysis.

RESULTS AND DISCUSSION

In the present work, nickel nanoparticles have been synthesized without adding any surfactant or capping agent by the polyol process. The XRD pattern of the as synthesized NiNP 12 is shown in Fig. 1 and there are much broader and less intense peaks, owing to particle size broadening, which occurs when a sample is made up of very small crystallites. Sharp peaks of nickel

were observed, which indicates the crystalline nature of the product. The characteristic peaks for NiNP, $2\theta = 39.18°$, 41.60°, 44.52° and 58.49°, corresponding to the Miller indices (010) (002) (011) and (012) were observed (JCPDS: 45 – 1027). These peaks revealed the presence of hexagonal nickel nanoparticles. The full width at half-maximum (FWHM) of the oriented peak can be used to calculate the average crystallite size of the nickel nanoparticles using the Scherrer's formula[12], ($D = 0.94\lambda / \beta\cos\theta$), where 0.94 is a constant value known as shape factor, λ is the wavelength, β is the full width at half maximum of the diffraction peaks and θ is the angle of diffraction. It reveals from the XRD pattern that the well defined sharp and broad peaks indicate the smaller crystallite size and also it is found to be in good agreement with the reported XRD pattern of Ni nanoparticles.

Table 1 - Experimental reaction conditions and the average crystallite size of the nickel nanoparticles (NiNPs) calculated from the XRD pattern

Sample	Concentration	Time duration (hours)	Temperature (^oC)	Average crystallite size (nm) from XRD data
EFFECT OF REACTION TIME				
NiNP 1	50 ml PEG-400+2g NA	4	220	24
NiNP 2	50 ml PEG-400+2gNA	5	220	12
NiNP 3	50 ml PEG-400+2g NA	6	220	14
NiNP 4	50 ml PEG-400+2g NA	7	220	15
EFFECT OF TEMPERATURE				
NiNP 5	50 ml PEG-400+2g NA	5	100	28
NiNP 6	50 ml PEG-400+2g NA	5	150	15
NiNP 7	50 ml PEG-400+2g NA	5	200	13
NiNP 8	50 ml PEG-400+2g NA	5	220	12
NiNP 9	50 ml PEG-400+2g NA	5	250	12
EFFECT OF CONCENTRATION				
NiNP 10	50 ml PEG-400+2g NA	5	220	12
NiNP 11	100 mlPEG-400+2g NA	5	220	14
NiNP 12	150 ml PEG-400+2g NA	5	220	15
NiNP 13	200 ml PEG-400+2g NA	5	220	37

In the study on the effect of reaction time on the size of the nickel nanoparticles, all the reaction conditions were kept constant except the time duration. The crystallite sizes of the NiNPs are shown in Table 1. The average crystallite size of the

NiNP 1 synthesized at 4 hours duration time is 24 nm and the size of NiNP 2 is 12 nm for 5 hours duration time. The crystallite size continues to increase along with duration of the reaction time. The crystallite size of the NiNP 3, synthesized at 6 hours duration is 14 nm and that of 7 hours duration is 15 nm. Hence it is concluded that the crystallite size of the NiNPs increases with increase in the reaction time. The particles begin to grow and agglomerate at long time durations, hence the crystallite size of the synthesized nickel nanoparticles increase along with the reaction time duration.

The reactions carried out to study the effect of reaction temperature on the size of the NiNPs, all the reaction conditions were kept constant except the reaction temperature. The reactions were carried out at 100^{o}C, 150^{o}C, 200^{o}C, 220^{o}C and 250^{o}C temperatures and the average crystallite sizes of the NiNPs calculated using Scherrer's formula are 28 nm, 15nm, 13nm, 12nm and 12 nm, respectively. Accordingly, the average crystallite sizes of the NiNPs decrease with increase in the reaction temperature. This is due to the fact that the chemical reduction of Ni^{2+} takes place effectively at higher temperature and it prevents the particle agglomeration, hence the average crystallite size decreases. And also some reactions were carried out by keeping all the reaction conditions constant except the concentration of the chemicals. The average crystallite size of the NiNPs increase as we increase the concentration of PEG 400. At high concentration of the PEG 400, the formed NiNPs begins to aggregate, hence the crystallite size increases.

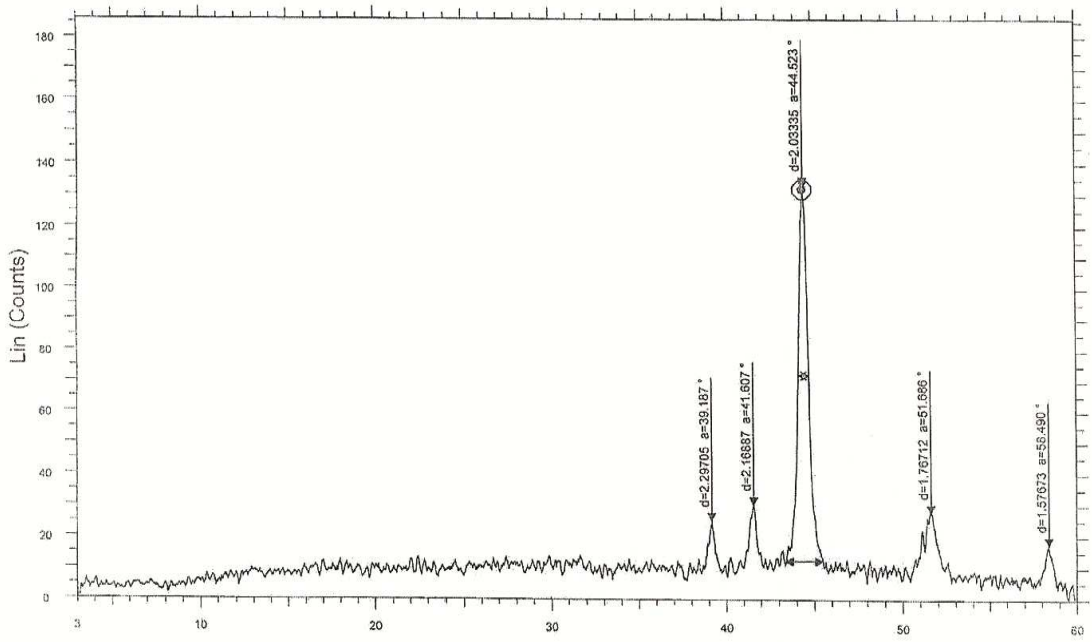

Fig. 1 XRD pattern of the as synthesized NiNP 12

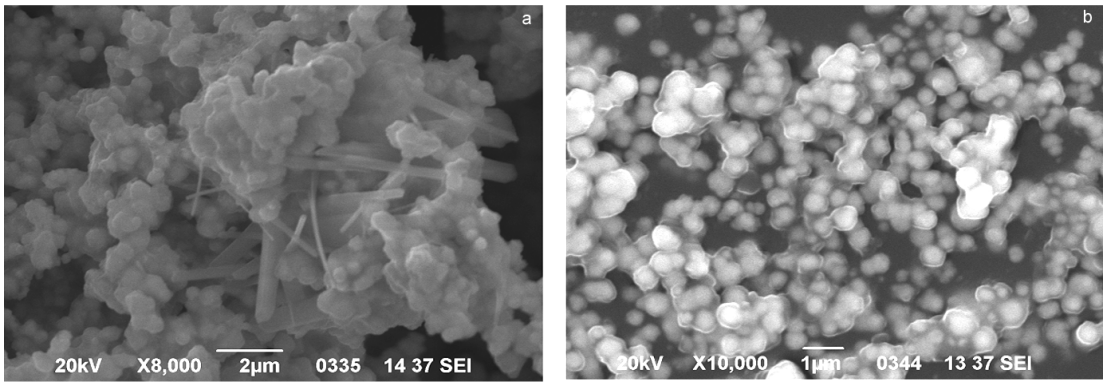

Fig. 2 SEM images of a) NiNP 5 showing rod like nanoparticles and b) NiNP 7

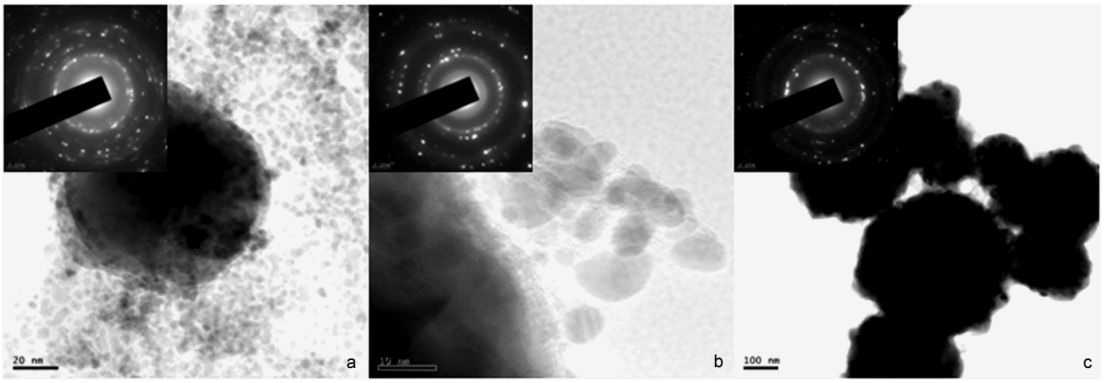

Fig. 3 HRTEM and corresponding SAED patterns of (a) NiNP 1; (b) NiNP 7; (c) NiNP 10

The microstructural characterization was investigated by using SEM and HRTEM, and shown in **Fig. 2** and **Fig. 3** respectively. SEM images of NiNP 5 (**Fig. 2a**) and NiNP 7 (**Fig. 2b**) shows a hexagonal like morphology of the nickel nanoparticles. In **Fig. 2a**, a rod like morphology was observed with different arrangement. In **Fig. 2b**, sphere like morphology was observed. HRTEM and Selected Area Electron Diffraction (SAED) (inset) patterns of synthesized NiNP 1, NiNP 7 and NiNP 10 are shown in **Fig. 3**(**a-c**) respectively. It is observed that the nanoparticles are encapsulated with the polymers in order to prevent agglomeration. It was observed that the temperature has an influence on the morphology of the nickel nanoparticles. A rod like nickel nanorods was formed at a temperature of 100°C, but at higher temperatures the rod like structures were disappeared.

Energy dispersive X-ray (EDAX) analysis on various regions confirmed the presence of nickel ,with energy bands centered at 7.5 and 8.3 kev (K lines) and 0.8 kev (L lines)[13]. The oxygen detected could be attributed to partial oxidation of the

nanoparticles during the handling of the sample or to the presence of some residual solvent. The EDAX spectrum of the NiNP 4 is shown in **Fig. 4**.

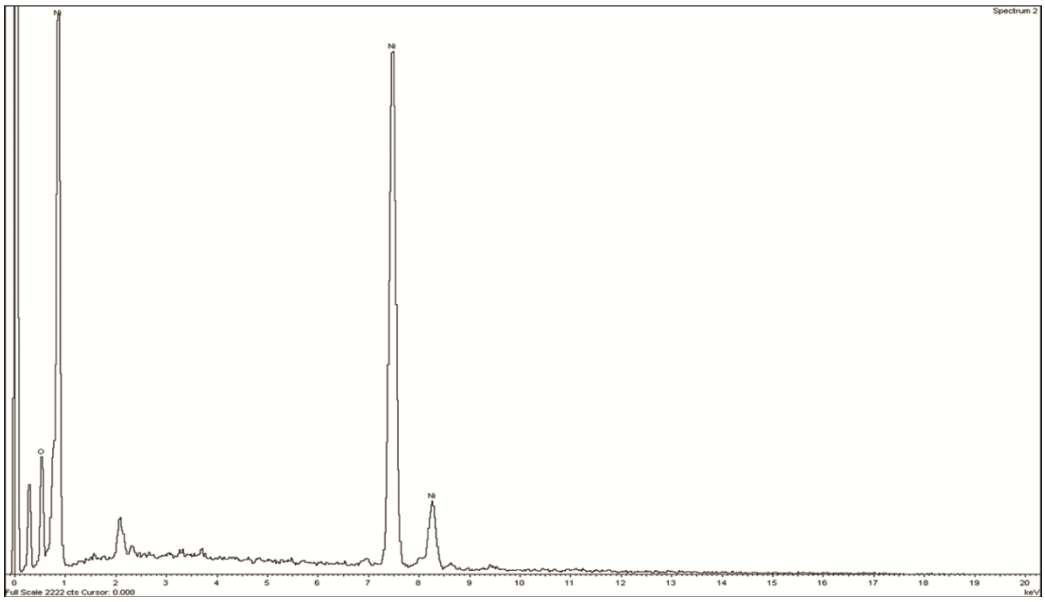

Fig. 4 - EDAX spectrum of NiNP 4

CONCLUSIONS

The NiNPs have been prepared using polyol process without adding any surfactant or capping agent and they were analyzed using XRD, SEM, TEM and EDAX analysis techniques. The crystallite size of the as synthesized NiNPs calculated using XRD pattern are in good agreement with the TEM results. The crystallite size of the NiNPs was reduced when we increase the time duration of the reaction. Nickel nanorods were observed at a temperature of 100°C, however, at higher temperatures the rod like structures were disappeared. It was observed that the crystallite size of the NiNPs decreases as we increase the reaction temperature and it is minimum at 220^{o} C. The concentration does have an influence on the size of the nanoparticles. Thus, controlling the concentration of constituent chemicals can control the size of the nickel nanoparticles synthesized.

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